

NASA/TM—2002-211549



Effects of Humidity on the Flow Characteristics of PS304 Plasma Spray Feedstock Powder Blend

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This report contains preliminary findings, subject to revision as analysis proceeds.

The Aerospace Propulsion and Power Program at NASA Glenn Research Center sponsored this work.

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Abstract

The effects of environmental humidity on the flow characteristics of PS304 feedstock have been investigated. Angular and spherical $\text{BaF}_2\text{-CaF}_2$ powder was fabricated by comminution and by atomization, respectively. The fluorides were added incrementally to the nichrome, chromia, and silver powders to produce PS304 feedstock. The powders were dried in a vacuum oven and cooled to room temperature under dry nitrogen. The flow of the powder was studied from 2 to 100 percent relative humidity (RH). The results suggest that the feedstock flow is slightly degraded with increasing humidity below 66 percent RH and is more affected above 66 percent RH. There was no flow above 88 percent RH. Narrower particle size distributions of the angular fluorides allowed flow up to 95 percent RH. These results offer guidance that enhances the commercial potential for this material system.

Introduction

It is common practice to dry plasma spray powders in order to enhance their flow properties, yet little experimental data is available on this issue. To better understand the flow behavior of PS304 versus the level of environmental humidity, flow tests were conducted in a controlled-humidity environment. PS304 is a plasma spray deposited coating for the reduction of friction and wear in turbomachinery applications [1-6]. The feedstock for this coating is a powder blend consisting of nichrome, chromia, silver and eutectic barium fluoride-calcium fluoride. Experience has indicated that the flow characteristics of the powder blend are highly dependent upon the morphology [7] and size [8] of the $\text{BaF}_2\text{-CaF}_2$ constituent. This paper reports the relationship between environmental humidity and the flowability of the PS304 feedstock powder. The effects of humidity were determined by comparing powder flow characteristics at a constant temperature while varying relative humidity (RH). The overall goal of this investigation was to better understand the effect of humidity on the flow characteristics of the feedstock with various $\text{BaF}_2\text{-CaF}_2$ particle sizes and morphologies in order to enhance the commercial potential of the PS304 coating.

The ability to understand and predict powder flow behavior is important in many manufacturing applications such as ceramics, powder metallurgy, powdered food, concrete and pharmaceuticals [9-13]. Therefore, this work is expected to help add insight to similar powder flow problems encountered in a wide variety of industries.

Background

The PS304 coating was developed at NASA Glenn Research Center [14] for the reduction of sliding friction and wear in turbomachinery applications at temperatures from sub-ambient up to 650 °C. The composition of this coating is 60wt% nichrome (80Ni-20Cr), 20wt% chromia (Cr_2O_3), 10wt% silver and 10wt% eutectic barium fluoride–calcium fluoride (70 BaF_2 -30 CaF_2). Nichrome serves as a binder and, along with chromia, provides wear resistance. Silver and BaF_2 - CaF_2 are solid lubricants at low temperature and high temperature, respectively. The coating is deposited on the sliding surfaces of the components by plasma spray.

In the plasma spray process particles of the deposition material are propelled by a carrier gas through a plasma flame, which is produced by the ionization of an inert gas [15]. The plasma heats the particles to a molten or plastic state. The semi-solid particles then strike the substrate and quickly solidify. The impact of subsequent particles increases the coating layer thickness. The feedstock is prepared in a powdered form to facilitate control of the rapid melting and re-solidification of the coating material. Where multi-component coating systems are to be deposited, the feedstock can be a powder blend composed of the different coating constituents.

In a preliminary study [16], it was found that intermittent clogging of the plasma spray powder feeding system was caused by the BaF_2 - CaF_2 particles. To examine the effect that the particle shape of the fluorides had on feedstock flowability, the fluorides were fabricated by comminution and by atomization. Comminution produced angular BaF_2 - CaF_2 particles and atomization produced spherical BaF_2 - CaF_2 particles. PS304 feedstock was prepared with each of these fluorides.

Approach

Flow of a powder system is affected by particle size and size distribution, particle shape and shape distribution, surface chemistry, density and the atmosphere surrounding the particles [17–27]. The effects of BaF_2 - CaF_2 particle shape, size and size distribution on the flowability of this powder blend are described elsewhere [7–8].

A powder will not flow under the influence of gravity when the forces between individual particles exceed the gravitational force acting on them. These forces can be described in terms of physical mechanisms known as interparticle friction, geometrical interlocking, and cohesion due to solid bridging, liquid bridging, and attractive electrostatic and van der Waals forces. The effects of interparticle friction on the PS304 feedstock powder have been investigated recently [7–8].

Geometrical interlocking is enabled by the mechanical interlocking of particles above the discharge orifice of the storage or feeding container, which blocks flow. Based on the work done by Langmaid and Rose [25], flow stoppage due to geometrical interlocking is highly unlikely for the PS304 feedstock powder due to the size of the particles in relationship to the orifice in the powder feed system. Based on their work, the critical diameter required to prevent flow interruption is four times the equivalent diameter of the particles in the powder system. The diameter of the outlet orifice of the Hall flowmeter (2.54mm), however, is more than twenty times the equivalent diameter of the largest particles in the PS304 powder blend (106 μm). Moreover, the outlet of the plasma spray system feed hopper is many times larger than that of the Hall funnel, which is true in general for gravity-fed hoppers.

Solid bridging due to solid state diffusion is also an improbable phenomenon due to the energy required for self diffusion of the materials used in the PS304 coating versus the operating temperatures in the plasma spray feed system. Of the remaining mechanisms, liquid bridging due to water meniscus forces is the focus of the current investigation.

When the vapor pressure p_o of condensed water on the surface of a particle approaches the partial pressure p of the water vapor in the air, the atmospheric humidity approaches saturation and liquid bridges may form at particle contact points [26]. A liquid bridge will be stable (i.e., it will not grow or evaporate) when $p_o = p$ or when

$$\frac{p}{p_o} = \exp\left(-\frac{M_w p_k}{\rho R T}\right)$$

where M_w is the molecular weight of water, p_k is the pressure due to the water meniscus force, ρ is the density of water, R is the gas constant for water vapor and T is the absolute temperature in Kelvin. At this point, adsorbed water on the surface of the particles begins to form liquid bridges as shown in Figure 1a. Due to the complexity of this system, a simplified case will be used to describe the mechanism more precisely.

A simplified liquid bridge between two idealized spherical particles is shown schematically in Figure 1b [27]. The tensile force due to the air-liquid interface is given by $2\pi bT$ where T is the surface tension of the liquid. The tensile force due to the pressure drop inside the liquid is given by $\pi b^2 P$, where P is the difference in pressure across the air-liquid interface. The pressure drop P can be calculated as the product of the liquid surface tension and the curvature of the liquid surface. From the figure, the principal radii of normal curvature are b and c . The curvature of the liquid surface is then $c^{-1} - b^{-1}$. Therefore, the total force acting between the two particles is

$$F = 2\pi bT + \pi b^2 T \left(\frac{1}{c} - \frac{1}{b} \right) = \pi bT \left(\frac{b+c}{c} \right).$$

The total force due to liquid bridging in a powder is the sum of all the individual particle-particle interactions.

The Hall flowmeter has been widely used as an indicator of the cohesive behavior of a powder system [28–33]. This instrument was a reliable indicator of the flowability of feedstock powder for the studied plasma spray system. The flow test measures the time required for a given amount of powder to exit a calibrated funnel, which is designated the *flow time*. As the attractive interparticle forces increase in a powder system, the flow time increases. The powders were first dried to remove adsorbed moisture. Comparing the flow times of powder with respect to controlled RH levels gave an assessment of the cohesiveness due to liquid bridging in the PS304 feedstock. Previous work [8] has also shown that the size and size distribution of the BaF_2 - CaF_2 particles can have a significant role in feedstock flow characteristics. Therefore, PS304 was also prepared with narrower BaF_2 - CaF_2 particle size distributions for controlled-humidity flow tests.

Experimental Procedure

Figure 2 shows photomicrographs of the constituents of PS304. The nichrome particles (Figure 2a) are 44 to 74 μm in size and have a rounded shape. The chromia particles (Figure 2b) are 30 to 44 μm in size and have an angular morphology. The spherical silver particles (Figure 2c) are 45 to 100 μm in size. The nichrome, chromia and silver powders are available commercially and their sizes and shapes were not modified in this study.

The BaF_2 - CaF_2 constituent was fabricated by two different techniques to obtain two distinct particle morphologies [7]. The angular fluorides, shown in Figure 3a, were fabricated by comminution of the fused eutectic. Figure 3b shows spherical fluorides, which were fabricated by gas atomization. An equal particle size distribution by mass of -140+325 mesh (45 to 106 μm) powders was prepared of comminuted

and of atomized $\text{BaF}_2\text{-CaF}_2$ powders, in addition to -140+170 mesh (90 to 106 μm) and -270+325 mesh (45 to 53 μm) comminuted fluorides.

The $\text{BaF}_2\text{-CaF}_2$ powders were classified by screening according to ASTM standard specification B 214-99. This procedure was performed using screens manufactured according to ASTM standard specification E-11. The screens were stacked vertically in order of coarsest mesh to finest mesh. The screen mesh sizes used were numbers 140, 170, 200, 230, 270, and 325. The screening instrument uses a vertically oscillating column of air and a combination of vertical and horizontal tappers to separate the particles according to size.

Two powder blends consisting of 60g nichrome, 20g chromia, 10g silver, and 10g of either angular or spherical fluorides were prepared by mixing the constituents together in a 125mL high density polyethylene bottle until the powder was well blended. Each powder was then spread evenly on the bottom half of a glass petrie dish and placed in a vacuum oven for 12 hours at 59mm Hg (absolute pressure) and 205 °C. The powders were then covered with the top half of the petri dish and transferred immediately to a glove box purged with dry nitrogen gas. The powders were allowed to cool to room temperature (~22 °C) in the nitrogen atmosphere. To control RH in the glove box, the nitrogen gas inlet was connected to a gas diffusion bubbler containing deionized water as shown schematically in figure 4. The powder samples were exposed to each humidity level for 2 hours prior to flow testing according to ASTM B 213-97. A digital stopwatch was used to measure the time it took for the entire 50g sample to exit the funnel to the nearest 0.1s. The average and standard deviation of 5 consecutive tests were reported.

Results and Discussion

The flow times of PS304 feedstock powders with 45 to 106 μm $\text{BaF}_2\text{-CaF}_2$ particle with respect to humidity are shown in figure 5. The plot shows that flow times for the PS304 feedstock with angular $\text{BaF}_2\text{-CaF}_2$ are higher than with spherical $\text{BaF}_2\text{-CaF}_2$. Three regions are labeled on figure 5 in order to discuss changes in flow behavior. In Region I (0 to 66 percent RH), flow is only slightly degraded with increasing humidity. Humidity has a greater effect on flow from 66 to about 88 percent RH (Region II) and in Region III (>88 percent RH) no flow was observed. The flow times increase with increasing relative humidity; this effect is less at lower humidity levels and increases at higher humidity, similar to results reported by Peterson and Small [31-32]. As shown in figure 6, PS304 feedstock with narrower size distribution 45 to 53 μm and 90 to 106 μm angular $\text{BaF}_2\text{-CaF}_2$ particle powders remained free flowing up to 95 percent RH.

The results for all four powder blends are summarized in table 1. The standard deviation of flow time measurements of powders containing spherical fluorides ranged from 0.1 to 0.2s, and from 0.1 to 0.4s for powders with angular fluorides. Due to the classification method, the surface to volume ratio of the angular and spherical fluorides was essentially the same. However, the angular fluorides may have larger contact areas than the spherical particles due to the relatively irregular surfaces of angular particles. Furthermore, the irregular shape of the angular fluorides makes the size of the contact area between adjacent particles much more random. An angular particle can contact an adjacent particle at a point, along a line, on a plane or on multiples and combinations of these contact modes. A spherical particle, on the other hand, is likely to have only point contact with adjacent particles. For this reason, it is believed that the variation in the measurements for powders with the spherical fluorides tends to be slightly lower than with angular fluorides. The significance of this finding for process engineering is that spherical fluorides may provide better control and repeatability of coating deposition over a typical range of laboratory atmospheric conditions.

Conclusions

The objective of this investigation was to study the effects of environmental humidity on the flow rate of PS304 feedstock under the influence of gravity. Based on the results, the following conclusions were made.

1. Humidity has a minor effect on the flowability of PS304 feedstock with 45 to 106 μ m fluorides from 2 to 66 percent RH.
2. Above 66 percent RH, humidity had a more detrimental effect on powder flow and above 88 percent RH no flow was observed for feedstock containing either angular or spherical 45 to 106 μ m fluorides
3. Feedstock flow could be obtained up to 95 percent RH using narrower size distributions of angular fluorides (45 to 53 μ m or 90 to 106 μ m).
4. The variation in the flow time measurement for feedstock using spherical fluorides tends to be lower than with angular fluorides.

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Table 1.—Summary of data from controlled humidity flow tests

BaF ₂ -CaF ₂ constituent size (shape)	Flow time increase, ^a %		Standard deviation of measurements	Maximum humidity for flow
	Region I	Region II		
45 to 106 μm (angular)	6.3	17	0.1 to 0.4s	88
45 to 106 μm (spherical)	3.3	25	0.1 to 0.2s	88
45 to 53 μm (angular)	8.6	9.7	0.1 to 0.3s	95
90 to 106 μm (angular)	10	8.6	0.1 to 0.2s	95

^aFor the feedstock with 45 to 53 μm and 90 to 106 μm BaF₂-CaF₂, Region I and Region II are from 2 to 70 percent and 70 to 91 percent RH, respectively.

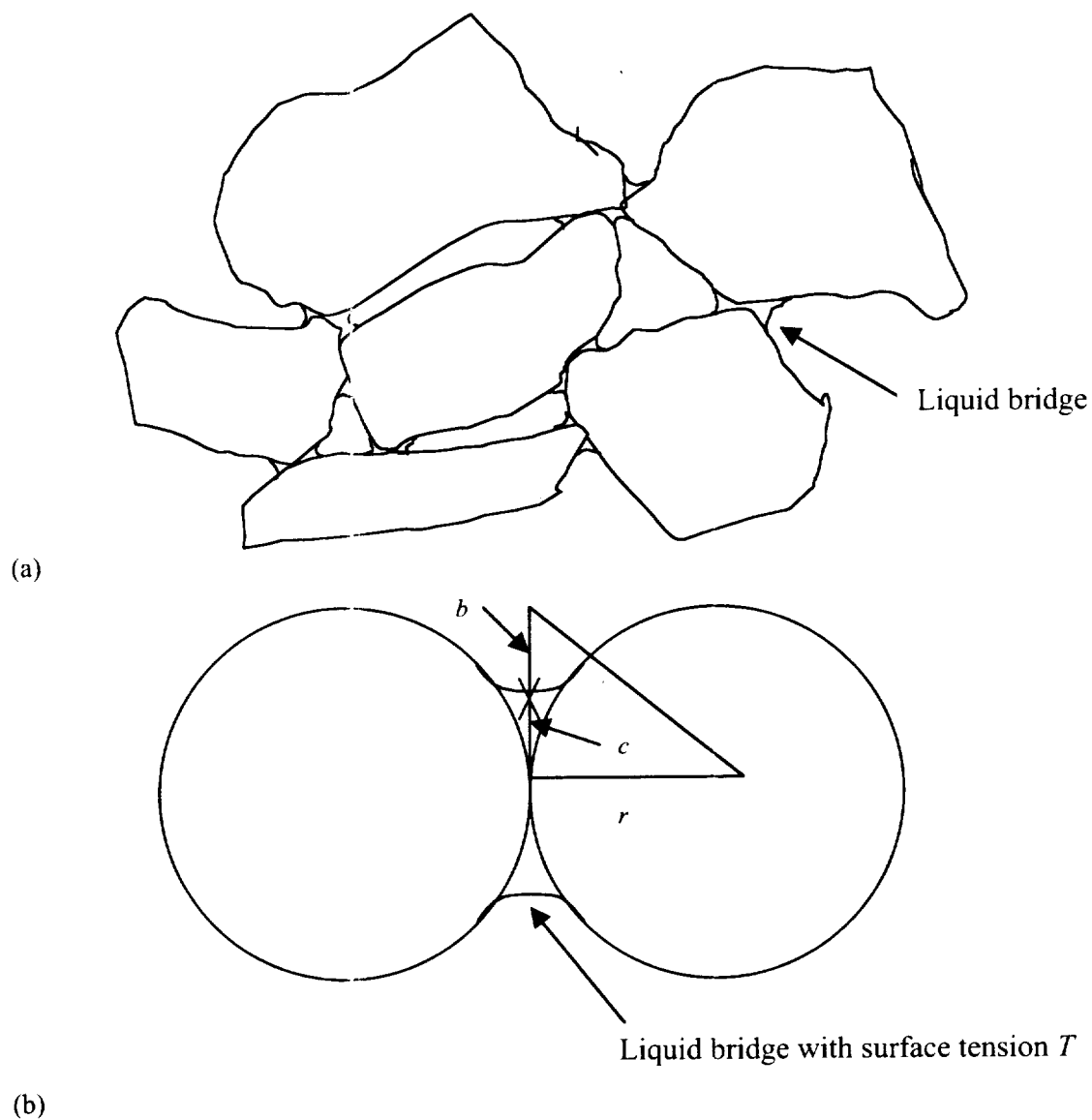
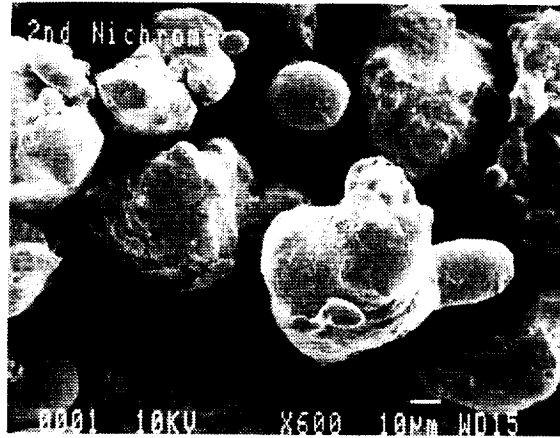
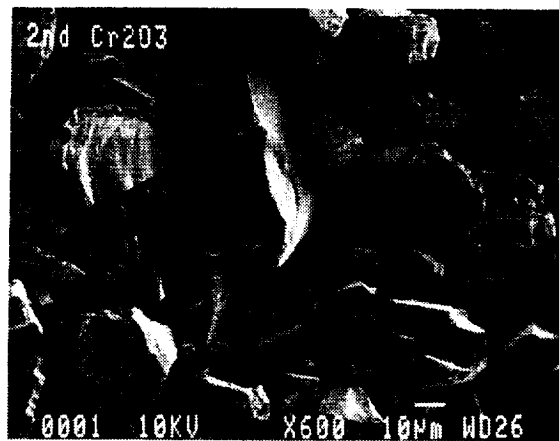


Figure 1.—Representation of liquid bridge formation between (a) Typical angular particles.
(b) Idealized spherical particles.



(a) Nichrome

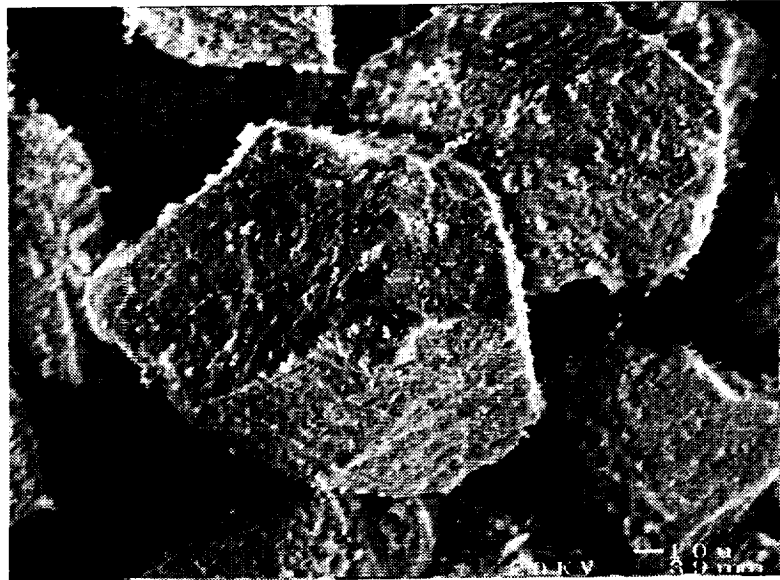


(b) Chromia

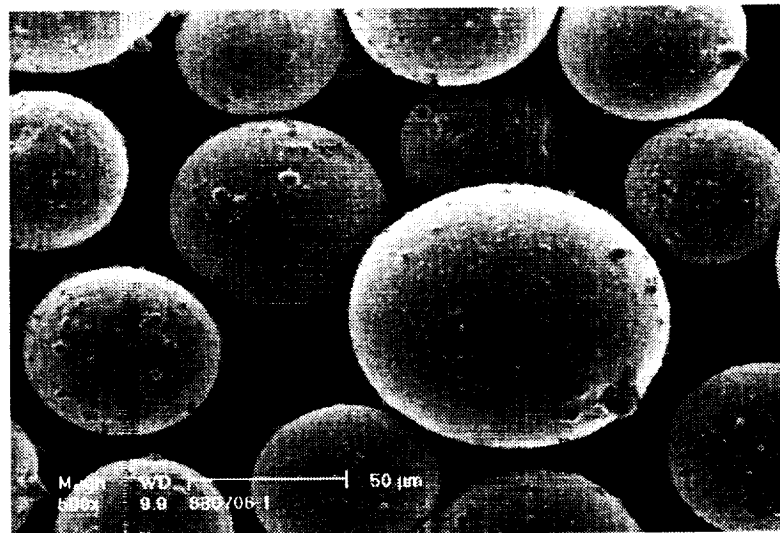


(c) Silver

Figure 2.—Nichrome, chromia, and silver constituents of PS304 feedstock powder (original magnification 600X).



(a)



(b)

Figure 3.—(a) Angular and (b) Spherical $\text{BaF}_2\text{-CaF}_2$ particles (original magnification 500X).

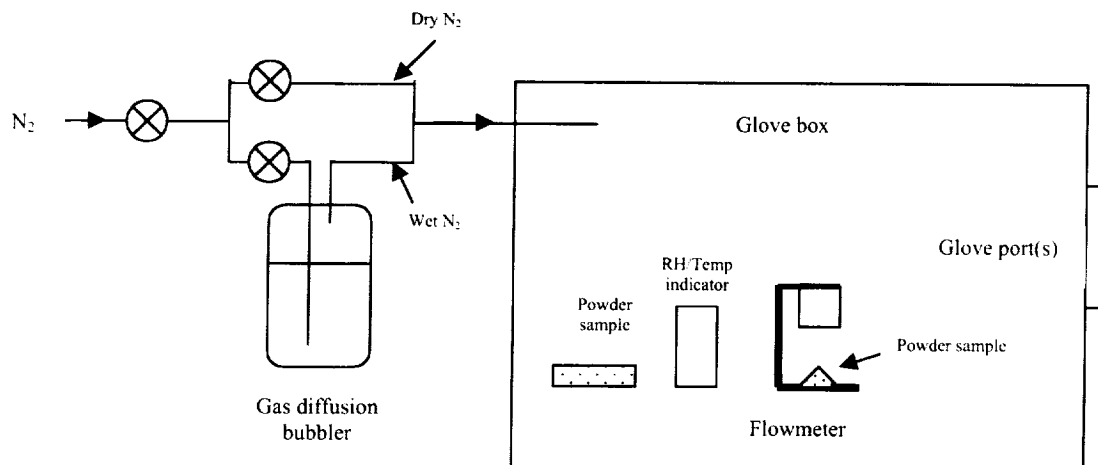


Figure 4.—Experimental setup for controlled humidity powder flow tests.

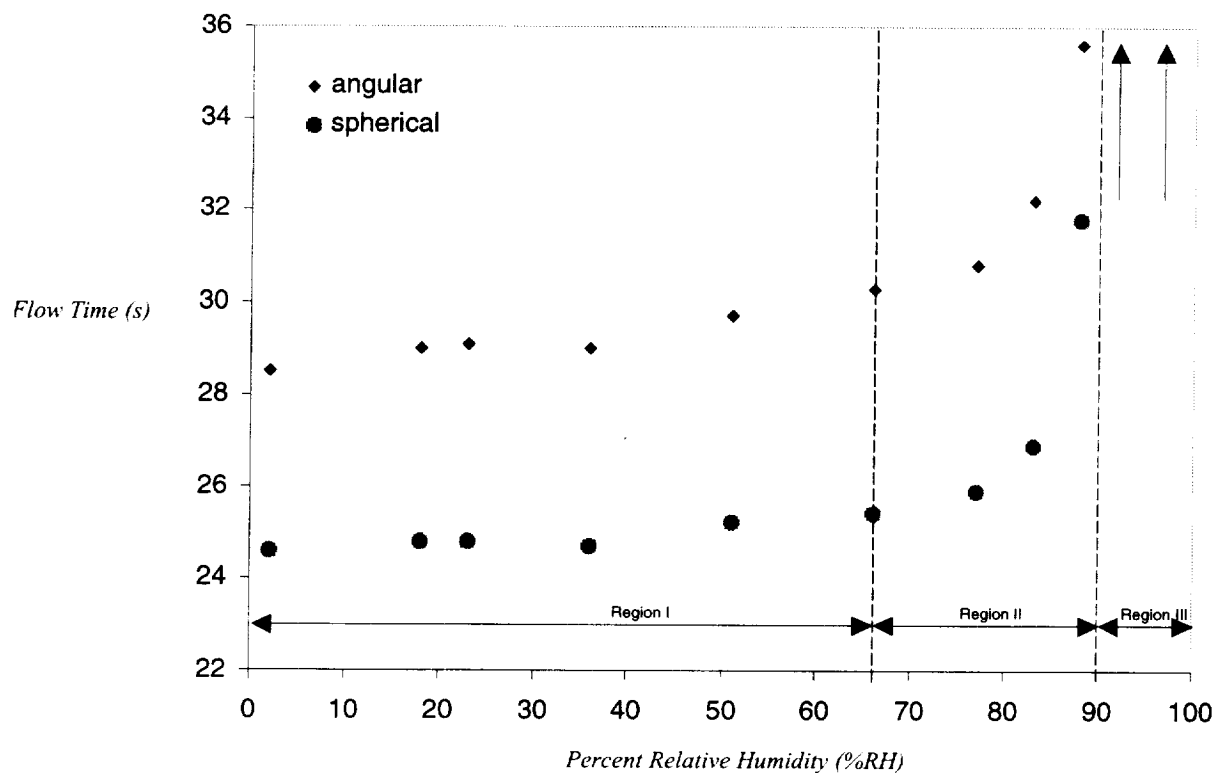


Figure 5.—Flow times of PS304 feedstock powders with 45 to 106 μ m angular and spherical BaF₂-CaF₂ particles as a function of percent relative humidity.

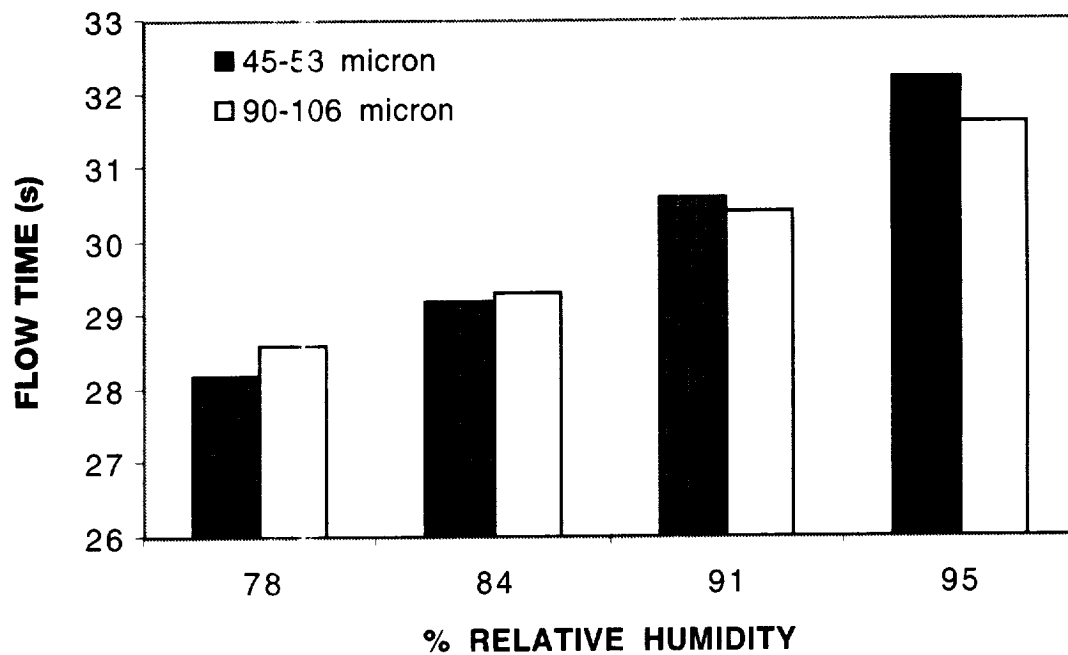


Figure 6.—Flow times of PS304 feedstock with angular 45 to 53 μ m and 90 to 106 μ m BaF₂-CaF₂ particles at higher humidity levels.

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1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE May 2002	3. REPORT TYPE AND DATES COVERED Technical Memorandum		
4. TITLE AND SUBTITLE Effects of Humidity on the Flow Characteristics of PS304 Plasma Spray Feedstock Powder Blend		5. FUNDING NUMBERS WU-708-18-13-00		
6. AUTHOR(S) Malcolm K. Stanford and Christopher DellaCorte				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration John H. Glenn Research Center at Lewis Field Cleveland, Ohio 44135-3191		8. PERFORMING ORGANIZATION REPORT NUMBER E-13313		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001		10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA TM-2002-211549		
11. SUPPLEMENTARY NOTES Malcolm K. Stanford, University of Dayton, Materials Engineering, 300 College Park, Dayton, Ohio 45469-0121, and Christopher DellaCorte, NASA Glenn Research Center. Responsible person, Christopher DellaCorte, organization code 5960, 216-433-6056.				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified - Unlimited Subject Category: 23 Available electronically at http://gltrs.grc.nasa.gov/GLTRS This publication is available from the NASA Center for AeroSpace Information, 301-621-0390.		12b. DISTRIBUTION CODE		
13. ABSTRACT (Maximum 200 words) The effects of environmental humidity on the flow characteristics of PS304 feedstock have been investigated. Angular and spherical BaF ₂ -CaF ₂ powder was fabricated by comminution and by atomization, respectively. The fluorides were added incrementally to the nichrome, chromia, and silver powders to produce PS304 feedstock. The powders were dried in a vacuum oven and cooled to room temperature under dry nitrogen. The flow of the powder was studied from 2 to 100 percent relative humidity (RH). The results suggest that the feedstock flow is slightly degraded with increasing humidity below 66 percent RH and is more affected above 66 percent RH. There was no flow above 88 percent RH. Narrower particle size distributions of the angular fluorides allowed flow up to 95 percent RH. These results offer guidance that enhances the commercial potential for this material system.				
14. SUBJECT TERMS Coatings; Lubrication; Solid lubricants; Powders; Plasma spray coatings; Relative humidity; Powder flowability; Powder feedstock			15. NUMBER OF PAGES 17	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT	